PATENT SPECIFICATION

NO DRAWINGS



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Inventors:—SABBAT JOHN STRIANSE and MARK ROY HAVASS.

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COMPLETE SPECIFICATION

A Cosmetic Preparation

We, YARDLEY AND COMPANY LIMITED, a British Company of London, England, do hereby declare the invention, for which we pray that a patent may be granted to us, 5 and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to hair grooming compositions, including hair fixature com-

(0) positions.

For many years compositions based on hydrocarbon oils and oil gels, such as pet-rolatum, have been sold for restoring oil to dry hair and scalp, to brighten the hair and 15 to impart to it a measure of manageability, Such compositions suffer from a variety of

disadvantages.

The oil gel types of hair grooming compositions, often called "pomades" or "solid 20 brilliantines" are most used for hair control; but they have poor lubricity, and poor spreadability, and thus are likely to leave a high degree of stickiness or greasiness on the hair, which is objectionable to many per-25 sons. Such compositions made by bodying mineral oil by use of known agents such as aluminium stearate or paraffin wax, are subject to separation of liquid oil (syneresis), friability of the gel structure, shrinkage with-30 in the jar, and poor spreadability. These characteristics have made such compositions less than satisfactory and have limited their

The heavier oils in liquid form also are 35 difficult to spread thinly and uniformly on the hair; whereas the thinner oils tend to run off the hands and hair and down onto one's face or neck or along one's wrist, onto sleeves or other parts of one's clothing.

The liquid oil compositions are also less than satisfactory in their grooming and fixative effects. The oil is not absorbed by the hair and, to the extent that it provides hair

control, it also causes an objectionable oiliness and apparent matting, "plastering 45 down" of the hair.

The present invention is based upon the discovery that desirable and effective hair grooming compositions can be made by at least partly dissolving polyamide material in 50 a liquid, oily, non-polar solvent consisting of or including a substance having a chain of at least 10 carbon atoms in its molecule, the polyamide material being a reaction product of an aliphatic poly-carboxylic acid and an 55 alkylene polyamine and having an average molecular weight between 2000 and 14000. Advantageously the polyamide material is of the type set forth in U.S. Patents Nos. 2,450,940 and 2,379,413, having an average 60 molecular weight between 2000 and 10000 and being reaction products of aliphatic dicarboxylic acids and di- or polyamino com-

The present invention provides a hair 65 grooming and hair treating composition which is free from the serious disadvantages of the prior compositions, and thus provides improvements in such compositions and in the art of hair grooming which have long 70 been sought, but had seemed unattainable.

The composition may include a cosolvent as well as the oily vehicle (the solvent). The cosolvent dissolves the resin and is miscible with the oil so as to bring the composition 75 into the form of a stable gel or suitably bodied or polymerized liquid. Other ingredients may be included for example, ordinary cosmetic diluents and ingredients. for example, scents and tinting colours.

These may be added to the oily vehicle non-polar materials solid at ambient temperature. The oily vehicle may include, for example, mineral oils which are advantageously of 50-70 Saybolt viscosity. Higher §5 viscosity oils have less compatibility with

[Price 4s. 6d.]

Price 33p

the polyamide material and give less clear

The cosolvents or coupling agents chosen are cosmetically acceptable compounds 5 which form with the resin a solution which is miscible with the oily vehicle. In general, the cosolvent is a substance in which both the resin and the oil are soluble. The most advantageous liquids as cosolvents are of the

10 class of fatty acids, alcohols and glycol esters having a hydrocarbon radical of the kind found in vegetable oils, most advantageously the hydrocarbon radical has a straight chain of 12-18 carbon atoms.

15 Typical examples of cosolvents are oleic acid, linoleic acid, mixtures of oleic and linoleic acids, diethanolamine linoleate, oleyl alcohol, propylene glycol mono laurate, propylene glycol di-laurate, propylene glycol

20 monomyristate, propylene glycol monooleate, lauryl lactate, myristyl lactate, methyl salicylate, castor oil, ethanol, isopropanol, di-butyl phthalate, di-butyl sebacate, and dioctyl sebacate, or mixtures thereof. How-

25 ever, some of the above cosolvents, for example propylene glycol mono laurate, can be used without the oily vehicle in which case they act as the oily liquid non-polar solvent.

30 The polyamide material as already indicated above, is advantageously a solid resinous, condensation product of an aliphatic dicarboxylic acid and a diamine (inclusive of compounds having at least one

35 alkylene and at least two amino groups, respectively) soluble in at least some organic

solvents. Suitable resins of this type are available commercially from General Mills, Inc., under the name "Versamid" (Registered Trade Mark), and from Olin-Mathison 40 Chemical Corp., under the name "Omamid" (Registered Trade Mark), for example Omamid "S" or Omamid "C". They are tough thermoplastic resins of the polyamide type insoluble in water and in many ketones 45. and ester solvents such as carboxylic acid amides, alcohols and chlorinated hydrocarbons, depending upon the particular acids and amines which have been used to form the resin and also upon the polymer length. 50 Monohydric alcohols, especially those having 3-8 carbon atoms, and chlorinated hydrocarbons are generally effective and hydrocarbons and ether solvents are in some cases effective per se and in some cases 55 effective only in mixtures with other solvents. Specifically, such solvents include namyl alcohol, iso amyl alcohol, benzene, iso butyl alcohol, ethyl alcohol, n-octyl alcohol, mono butyl ether of ethylene glycol, mono 60 ethyl ether of ethylene glycol, n-propyl alcohol, iso propyl alcohol, turpentine, xylene and mixtures thereof. Chloroform, methylene chloride, turpentine and xylene, even though effective for solvent purposes, are not 65 recommended for hair grooming cosmetics. because of odour. In general, solubility is low or absent with simple hydrocarbons, but as indicated above, they may be useful in mixtures with other solvents.

Other properties of these resins appear as follows:—

				Versamid	!		Om	amid
	Resin type	900	930	940	950	100	C	S
7 5	Specific gravity	.98	.98	.98	.98	.98	.9799	.9496
	†Colour, Gardner	12	12	12	12	12	12- 14	11- 12
	Melting point °C	180-190	105-115	105-115	43-55	43-55		
	Softening point °C (ring							
	and ball) Viscosity						90-100	100-120
80	‡Brookfield at 150°C							
	Solid No. 2 Spindle		30-45	15-30	7-15	10-15	32- 52	55 - 75
	Acid value	_	_	_			12	12
	Amine value*	3	3	3	3	83-93		

*Amine value is the weight of KOH, in milligrams, equivalent to the free amine groups in one gram of the resin.

†Gardner Colour Scale (Transparent Liquids) Ref: American Society for Testing of Materials (Standards) Part 21, 1964.

†Brookfield viscometer data, Ref: American Society for Testing of Materials (Standards), Part 26, 1964.

90 "Versamid" polyamide resins are thermoplastic condensation products of polymerized linoleic acid with various polyamine compounds such as ethylene diamine, and diethylene triamine. Resins of average 95 molecular weights of 5000-10000 have been found best for the present invention. These resins are commercially available in hard.

brittle resin (No. 900) of melting point 180-190°C., tough flexible resins (No. 930 and 100 940) melting points 105-115°C., and in semisolid, soft tacky resin (No. 100), melting point 43-55°C., and with some wax added (No. 950), which results in some turbidity in the final product. These resins are compatible with each other so that by blending 105 them almost any desired properties in the aforesaid melting point range can be attained.

The tendency to syneresis of the oilpolyamide-cosolvent gels can be controlled 110 1,117,129

	by use of amides having 12 to 18 carbon	2. Simple system for clear gel:		
	atoms in their molecules, for example di-	Polyamide 8000 average MW	5.00	
	ethanolamides fulfilling this condition and/	Propylene glycol mono laurate	70.00	
	or by curing the gels by holding them at	Light mineral oil	25.00	
5	temperatures between their melting point			<i>7</i> 0
	and the ambient temperature.		100.00	
	Hair treating gels are prepared by dis-	3. Simple system for cloudy gel:		
	solving the polyamide resins in the hot or-	Polyamide 8000 MW (average)	5.00	
	ganic system comprising the oily vehicle and	Propylene glycol mono laurate	47.50	
10	the cosolvents (if included). Upon cooling,	Light mineral oil	47.50	75
	a gel structure is produced, and the pro-		100.00	
	perties will vary depending upon the amount	4.60 1 4 1 1	100.00	
	of resin employed, the composition and	4. Simple system including large		
	molecular weight of the resin, and the com-	percentage of mineral oil		40
12	patibility and solubility of the resin in the	(cloudy soft gel):	0.00	80
•	vehicle chosen. The solubility of the poly-	Polyamide 8000 MW (average)	2.00	
	amide resin in preferred solvent systems in-	Oleic Acid	13.00	
	creases with temperature. Whenever the	Light mineral oil	85.00	
20	solubility limits of the polyamide resin in a		100.00	95
20	particular solvent system are exceeded, a gel results which is thermally and mechanically	5. Simple system for clear gel	100.00	85
	reversible. Gels can be produced of a con-	using a blend of polyamide		
	sistency from a soft jelly-like to a firm rigid	resins:		
	structure, or of grainy, "crystal-like" struc-	Polyamide 8000 MW (average)	2.50	
25	ture or an amorphous, smooth glass-like	Polyamide 5000 MW (average)	2.50	9 0.
بيد	structure.	Propylene glycol mono laurate	70.00	JM.
	Mixing at high temperatures, e.g. in the	Light mineral oil	25.00	
	range 100-115°C may cause crystallization,	Light Himothi Off	25.00	
	whereas if the composition is mixed below		100.00	
30	100°C and poured into moulds or jars at	The light mineral oil referred		95
	about 55°C and then kept for a substantial	example is Marcol GX available		
	curing time, e.g. 1-6 days at an intermediate	Standard Oil Co. The use of oth		
	temperature, e.g. 37°C±2°C., a smoother	oils in many systems produce		
	structure and freedom from syneresis are	cloudy gels. However, these oth		
35	attained. (See Example 9 below).	oils can be made to give crystal		
	Such compositions are economical, thixo-	tems by rebalancing the cosolven		
	tropic and less subject to syneresis than the	The gels of Examples 2 to 5 ma		
	brilliantine type compositions having a metal	by heating the oily solvent and th		
	soap, such as aluminium stearate, as the	to slightly above the melting pe		
40	gelling agent, in mineral oil.	polyamide which is then introduc		
	It is an important advantage of the inven-	mixed solvents with agitation unt	il the mass	S
	tion that crystal clear gels can be made, al-	is homogeneous. The temperat	ure is re	•
	though opaque or cloudy gels are also with-	duced to below 100°C and variou		
45	in the broader scope of the invention. Such	are then introduced into the mas		
43	gels, whether crystal clear or non-clear, pro- vide a thixotropic, non-greasy solid gel,	ring. It is then cured and filled in containers.	ito suitadie	c 110
	which is liquefied quickly to a fluid by rub-	A further improvement has be	en attaine	4
	bing and is thus readily spread on and	by using appropriate mixtures of		
	throughout the hair leaving the hair con-	of the resin and mineral oil. This		
50	trolled and well groomed but with a natural	crease of the mineral oil content		
	soft appearance free from the "plastered-	sponding decrease in cost, while		
	down" look and greasiness which have made	higher temperature stability and	clarity o	î
	most hair dressing compositions unaccept-	the gel:		
	able to many persons.	Example 6 — A clear gel system	using two	0
5.		cosolvents:		120
	embodying the present invention which are		Percentag	
	suitable for hair grooming products:—		by weigh	
	Examples of Gels % By Weight	Polyamide 8000 average MW	5.00	
_	1. Clear tacky and substantially	Propylene glycol mono laurate	19.10	
60	_	Oleic Acid	10.90	125
	Polyamide 5000-8000 average	Light mineral oil	64.00	
	MW 5.00	Perfume	1.00	
	Castor Oil 95.00		100.00	
	100.00		100.00	***
6	5 100.00	•	,	130

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3

5 1	by use of amides having 12 to 18 carbon atoms in their molecules, for example distribution and/or by curing the gels by holding them at temperatures between their melting point and the ambient temperature.	2. Simple system for clear gel: Polyamide 8000 average MW Propylene glycol mono laurate Light mineral oil	5.00 70.00 25.00	70
10	Hair treating gels are prepared by dissolving the polyamide resins in the hot organic system comprising the oily vehicle and the cosolvents (if included). Upon cooling, a gel structure is produced, and the properties will vary depending upon the amount	3. Simple system for cloudy gel: Polyamide 8000 MW (average) Propylene glycol mono laurate Light mineral oil	5.00 47.50 47.50 100.00	75
15	of resin employed, the composition and molecular weight of the resin, and the compatibility and solubility of the resin in the vehicle chosen. The solubility of the polyamide resin in preferred solvent systems increases with temperature. Whenever the	4. Simple system including large percentage of mineral oil (cloudy soft gel): Polyamide 8000 MW (average) Oleic Acid Light mineral oil	2.00 13.00 85.00	80
	solubility limits of the polyamide resin in a particular solvent system are exceeded, a gel results which is thermally and mechanically reversible. Gels can be produced of a con-	5. Simple system for clear gel using a blend of polyamide	100.00	85
25	sistency from a soft jelly-like to a firm rigid structure, or of grainy, "crystal-like" structure or an amorphous, smooth glass-like structure. Mixing at high temperatures, e.g. in the	resins: Polyamide 8000 MW (average) Polyamide 5000 MW (average) Propylene glycol mono laurate Light mineral oil	2.50 2.50 70.00 25.00	9 0.
30	range 100-115°C may cause crystallization, whereas if the composition is mixed below 100°C and poured into moulds or jars at about 55°C and then kept for a substantial curing time, e.g. 1-6 days at an intermediate temperature, e.g. 37°C±2°C., a smoother	The light mineral oil referred example is Marcol GX available Standard Oil Co. The use of oth oils in many systems produces	100.00 to in this from Esse er minera	1
35	structure and freedom from syneresis are attained. (See Example 9 below). Such compositions are economical, thixotropic and less subject to syneresis than the	cloudy gels. However, these oth oils can be made to give crystal tems by rebalancing the cosolvent The gels of Examples 2 to 5 ma	er minera clear sys fraction.	1 - 100
40	brilliantine type compositions having a metal soap, such as aluminium stearate, as the gelling agent, in mineral oil. It is an important advantage of the invention that crystal clear gels can be made, al-	by heating the oily solvent and the to slightly above the melting po- polyamide which is then introduce mixed solvents with agitation unti- is homogeneous. The temperate	cosolven bint of the ed into the I the mas are is re	t e c 105 s
45	though opaque or cloudy gels are also with- in the broader scope of the invention. Such gels, whether crystal clear or non-clear, pro- vide a thixotropic, non-greasy solid gel, which is liquefied quickly to a fluid by rub-	duced to below 100°C and various are then introduced into the massering. It is then cured and filled in containers. A further improvement has been	with stir to suitable n attaine	e fio d
50	bing and is thus readily spread on and throughout the hair leaving the hair controlled and well groomed but with a natural soft appearance free from the "plastered-down" look and greasiness which have made	by using appropriate mixtures of of the resin and mineral oil. This crease of the mineral oil content sponding decrease in cost, while higher temperature stability and	allows in with corre preservin	- - 115 g
55	most hair dressing compositions unacceptable to many persons. Following are examples of compositions embodying the present invention which are suitable for hair grooming products:—	the gel: Example 6 — A clear gel system cosolvents:	using tw Percentag by weigh	120 re
60	Polyamide 5000-8000 average MW 5.00	Polyamide 8000 average MW Propylene glycol mono laurate Oleic Acid Light mineral oil Perfume	5.00 19.10 10.90 64.00 1.00	125
65	Castor Oil 95.00 100.00	* (4)	100.00	130

	The mineral oil can be used i	n an amount
	from 1-80% by weight of the polyamide in an amount from	ger, and the
	weight, (advantageously 2-10%	by residet
_	but in general the use of higher	ne weight)
Э	of oil leads to some deterioration	proportions on of the gel
	structure and its thermal stabi	lity If the
	product is subjected to long sto	race at am-
	product is subjected to long sto bient temperatures, or higher, ev	en the com-
10	positions made as above may	show some
	tendency to syneresis. When an	y separation
	becomes evident, even though	the product
	still preserves its excellent hai	r grooming
	properties, the customer may as	sume that it
15	is "spoiled." In such cases, spe	cial precau-
	tions should be taken.	
	This problem can be contro	olled and a
	gelled thixotropic hair groom	composition
	which does not break down or se	parate, even
20.	after long periods of storage at a	moient tem-
	peratures, can be provided. This	is attained
	by including in the composistabilizer, an amide having a	tion as a
	least 10 carbon atoms in its mol	emie The
25	following amides are advant	ageous as
20	stabilisers:	
	Lauryl diethanolamide	
	Stearyl diethanolamide	
	Oleyí diethanolamide	
30	Lauryl diethanolamide-Ethoxyl	ated Nonyl
	Phenol Adduct	
	Linoleyl diethanolamide	
	Coconut oil fatty acids diethan	nolamide
	Coconut oil diethanolamide	•
.35	Tallow fatty acid diethanolami	ae _::_
	Soy bean fatty acid diethanolas Kritchevsky Condensates such as	· — mige
	Coconut Oil-Diethanolamine	.—
	Condensate and	
40	Lauric Acid-Diethanolamine	
70	Condensate.	
	(Kritchevsky Condensates are)	products of
1	the condensation of polyalkyloi a	mines with
1	fatty acids or glycerides thereof,	said acids
	having 12-14 carbon atoms in t	heir mole-
(cules).	
4	As an illustration of the mann	er of using
	such stabilisers the following e	xampie is
	given: E <i>xample 7</i> — Stabilised clear gel s	meterne (at
JU 1	ambient temperature	systems (at
	%	By Weight
	Polyamide 8000 average MW	5.00
	Propylene glycol mono laurate	13.40
55	Lauryl diethanolamide	5.70
	Oleic acid	10.90
	Light mineral oil	64.00
	Perfume	1.00
œ		100.00
<i>6</i> 0		100.00
	Protection against syneresis can	he pained
	Trameron agamer shiretesis can	oc gamen

or extended by curing the gel at a constant temperature between the melting point of the gel and ambient temperature. After such water 67.00

Perfume 1.00

In the above example, propylene glycol mono laurate again constitutes the liquid, 130

curing our gelled compositions are protected against syneresis at considerably higher temperatures.

The combination of the polyamide resin and oil with cosolvent can also be used for 70 its extraordinary hair grooming and fixative effect in other than gel form. Examples of such are:

Example 8—Spray Aerosol Polyamide 8000 average MW Ethyl alcohol (specially	% By 7 5.00	Veight	75
denatured alcohol No. 40 anhydrous) Lanolin oil	80.00 1.00	35%	
Propylene glycol mono laurate Perfume			804

Propellant 11 (Trichloro mono-fluoro methane)

Propellant 12 (Dichloro di-fluoro methane)

50.00

65 % 85

The above is an example of the case mentioned previously where propylene glycol mono-laurate acts as the liquid, oily non-polar solvent rather than as a cosolvent. Instead of lanolin oil, other lanolin derivatives 95 which are compatible with the system may

be used.

Example 9 — Clear Liquid Brilliantine
Thixotropic Liquid type % By Weight
Polyamide 8000 average MW 5.00 100
Propylene glycol mono laurate 84.00
Light mineral oil 10.00
Perfume 1.00

Free Flowing Liquid type
Polyamide 8000 average MW 5.00 105
Oleic Acid 84.00
Light mineral oil 10.00
Prefume 1.00

Perfume 1.00

Example No. 10 — Alcoholic Liquid Hair

Groom 100

Parts

GIOUH		110
	Parts	
	by Weight	t
Polyamide 8000 average MW	2.00	
Light mineral oil	43.70	
Propylene glycol mono laurate	14.00	115
Oleic Acid	7.30	
Ethyl alcohol (specially	7,100	
denatured alcohol No. 40.		-
anhydrous)	32.00	
Perfume	1.00	120
Example 11 - Emulsified Cre		
Groom		
Polyamide 8000 average MW	5.00	
Propylene glycol mono laurate	15.00	
Stearic acid	10.00	125
Triethanolamine	2.00	
Water	67.00	
Perfume	1.00	
In the above example, propyle		
		-

oily, non-polar solvent as in Example 8.

The above emulsion is an example of an anionic type system. To those skilled in the art of emulsion making, it is readily understood that similar systems can be produced using non-ionic and cationic emulsifiers or combinations of both.

The use of the above formulations, comprising the polyamide resin together with the 10 other ingredients, results in more enhanced hair grooming efficiency and produces a greater brilliance than that obtained with formulations of the conventional type.

Hair grooming properties of polyamides 15 in gel systems were substantiated by halfhead experiments, as described below:—

A gob of a clear gel about the size of a finger nail was weighed and liquefied by rubbing in the palm of the hand and applied to hair on half of one's head. An equivalent weight of the same formulation, but without the polyamide contained therein, was applied to the other half of the same head. Both halves were combed identically and the halves were compared for brilliance, grooming qualities, and hair fixative properties. The results indicated that the half-head containing the polyamide was superior in the aforementioned qualities. The preceding test was more demonstrative when hair switches of identical hair were used in place of the half-heads.

It should be recognised that in addition 35 to improving hair grooming properties, the use of the polyamide resins, as a gelling agent for solid brilliantines, is also new.

WHAT WE CLAIM IS:

1. A hair grooming composition compris-4() ing a polyamide material at least partly dissolved in a liquid, oily, non-polar solvent consisting of or including a substance having a chain of at least 10 carbon atoms in its molecule, the polyamide material being a

45 reaction product of an aliphatic polycarboxylic acid and an alkylene polyamine and having an average molecular weight between 2000 and 14000.

2. A hair grooming composition as 50 claimed in claim 1 in which the polyamide material is solid at ambient temperatures.

3. A hair grooming composition as claimed in claim 2 including a cosolvent which forms with the polyamide a solution 55 which is miscible with the liquid, oily solvent.

4. A hair grooming composition as claimed in claim 2 or 3 in which the liquid,

oily solvent has a viscosity between 50 and 70 Saybolt.

5. A hair grooming composition as claimed in claim 2, 3 or 4 which contains from 1% to 40% by weight of the polyamide material and from 1% to 80% by weight of the liquid oily solvent.

6. A hair grooming composition as claimed in claim 2, 3, 4 or 5 which contains from 2% to 10% by weight of the polyamide material.

7. A hair grooming composition as 70 claimed in any of the claims 2 to 6 in which there is incorporated a stabiliser which is an amide having a chain of at least 10 carbon atoms in its molecule.

8. A hair grooming composition as 75 claimed in claim 7 and which has been cured at a temperature between the melting point of the composition and ambient temperature.

9. A hair grooming composition as claimed in any one of claims 2 to 8 in which 80 the said polycarboxylic acid is polymerized linoleic acid.

10. A hair grooming composition as claimed in any one of claims 2 to 9 in which the average molecular weight of the poly- 85 amide material is from 5000 to 10000.

11. A hair grooming composition as claimed in claim 7 or 8 in which the said stabiliser is a diethanolamide having 12 to 18 carbon atoms in its molecule.

12. A hair grooming composition as claimed in any one of the preceding claims including a perfume.

13. A method of grooming hair comprising the step of applying a hair grooming 95 composition according to any one of the preceding claims to the hair.

14. A method of preparing a hair grooming composition according to claim 1 comprising the steps of forming a mixture of the 100 polyamide material in the oily liquid non-polar solvent, and at least partially dissolving said polyamide in the solvent by applying heat to said solvent either before or after addition of said polyamide.

15. A hair grooming composition substantially as described in any one of the examples hereinbefore set forth.

16. A method of preparing a hair grooming composition substantially as herein- 110 before described.

For the Applicants,
WILSON, GUNN & ELLIS,
Chartered Patent Agents,
57 Market Street,
Manchester, 1.

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